Report of the experiment 08-02-618: Thermal stability and bcc-fcc phase transition upon heating treatment of Fe nanoclusters in silica: in-situ grazing incidence XRD investigation.

The aim of the experiment was to investigate the thermal stability of bcc Fe clusters in silica obtained by ion implantation, in particular to measure the bcc->fcc transition that in the bulk Fe occurs at 912°C. Different silica slides were implanted with various Fe fluences, in order to obtain bcc Fe clusters with different initial average size. Then each sample was heated in vacuum and the grazing incidence x-ray diffraction patter was monitored. Unfortunately, we encountered a major problem that prevents us to conclude the experiment successfully. In fact, the diffraction pattern from the Fe clusters, if taken in grazing incidence mode, was 'double', in the sense that besides the bcc pattern another set of peaks appeared, corresponding to the bcc set, just translated in another position. The beamtime was then used to investigate the problem that has been demonstrated to depend on the second mirror, used for the first time in a grazing incidence x-ray diffraction experiment. In figure the diffraction pattern from one of our sample is shown, upon radial integration of the 2D image recorded with the imaging plate: in the inset the whole pattern is visible, while in the large image the second set of peaks is clearly visible. Unfortunately, the main peak of the fcc phase should appear upon heating just in correspondence of the first peak of the second set (located in the graphic at about $2\theta \sim 19$ deg). Further work on the beamline is needed to solve the problem. Another possibility is



to perform the experiment without mirrors (but loosing at least a factor of 3 in the x-ray flux), as this configuration has been already successfully tested in previous experiments.